Operator's Handbook

Cryojet

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Contents

Warnings

Before you attempt to install or operate this equipment for the first time, please make sure that you are aware of the precautions that you must take to ensure your own safety. The booklet “Safety Matters”, supplied with this manual gives advice.

1 Quick User Guide

A) If the system has not been installed and set up, see section 3.

B) Check there is at least 10 cm of liquid nitrogen in the dewar. Never allow the dewar to become empty when the Cryojet is running.

C) Switch on the controller. See "Default conditions on startup" below.

D) Set the temperature.

E) Set the flow rates.
Suggested sample flow rate for 100 K: 6 l/min
Suggested sample flow rate for 90 K: 10 l/min
Suggested shield flow rate: 5 l/min.
For a fast cool-down, set the sample flow rate to 10 l/min initially, and then reduce it to the desired value when the temperature is close to the set temperature.

F) Turn the automatic temperature control ON. The Cryojet will now cool to the set temperature. (To select Automatic mode, the sample flow must be at least 2l/min. If a flow has been selected but after a couple of minutes no flow can be felt, switch the temperature control **off** and see section 9.)

G) Align the nozzle with the anticipated sample position: see "Alignment of Nozzle" below.

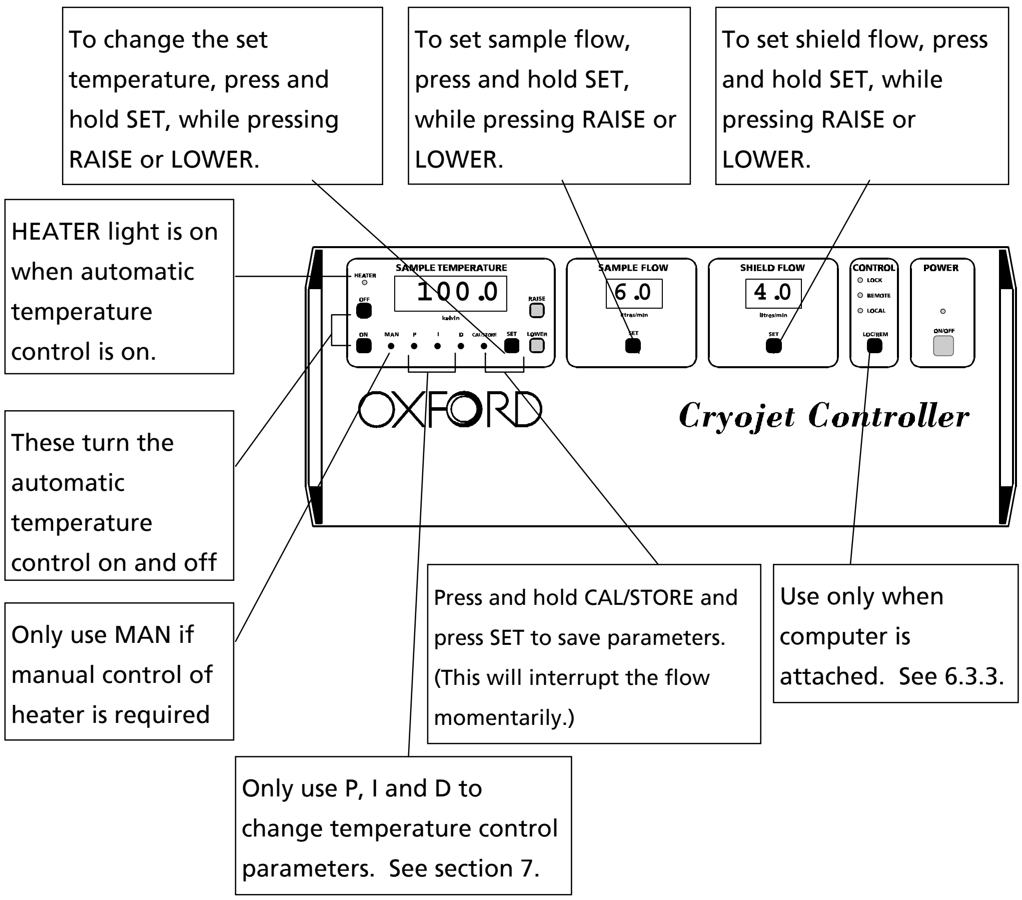

Figure 1 Main features of Cryojet controller

Keep the plastic cap and the pressure relief valve in place on the dewar top fitting, to prevent the ports becoming blocked by ice.

Alignment of Nozzle

The nozzle should be placed as close to the sample as possible to prevent icing - 5 mm is recommended.

The jet should be centred on the sample. One way to do this is to use the Oxford Instruments alignment tip. Slide the device into the nozzle, and tighten the knob to clamp it in place. Place a sample mount with no sample on the goniometer. Adjust the nozzle so that the sharp tip is almost touching the place where the sample would be. Remove the sample mount. Loosen the knob and remove the alignment tip. Then place the sample on the goniometer. If required, move the coldhead forward using the micrometer.

Using the dipstick to measure the level of liquid nitrogen

Slowly lower the dipstick into the dewar through the fill port until it hits the bottom, and wait a couple of seconds. Then pull it out, and wait a few seconds for frost to form on the surface. The length of the frosted section is the depth of liquid in the dewar. Do not touch the cold part of the dipstick with your bare hands.

Warming up the system

The system should be switched off whenever it is not in use. To prevent the remote possibility of ice or water building up in the coldhead (which is designed to prevent this happening), either

(a) block the inner nozzle with a rubber stopper. The cold head can then be left to warm up naturally. Or

(b) set the temperature to 300 K and allow the displayed temperature to reach room temperature before switching off. To speed this up, reduce the sample flow to about 3 l/min.

Default conditions on start-up

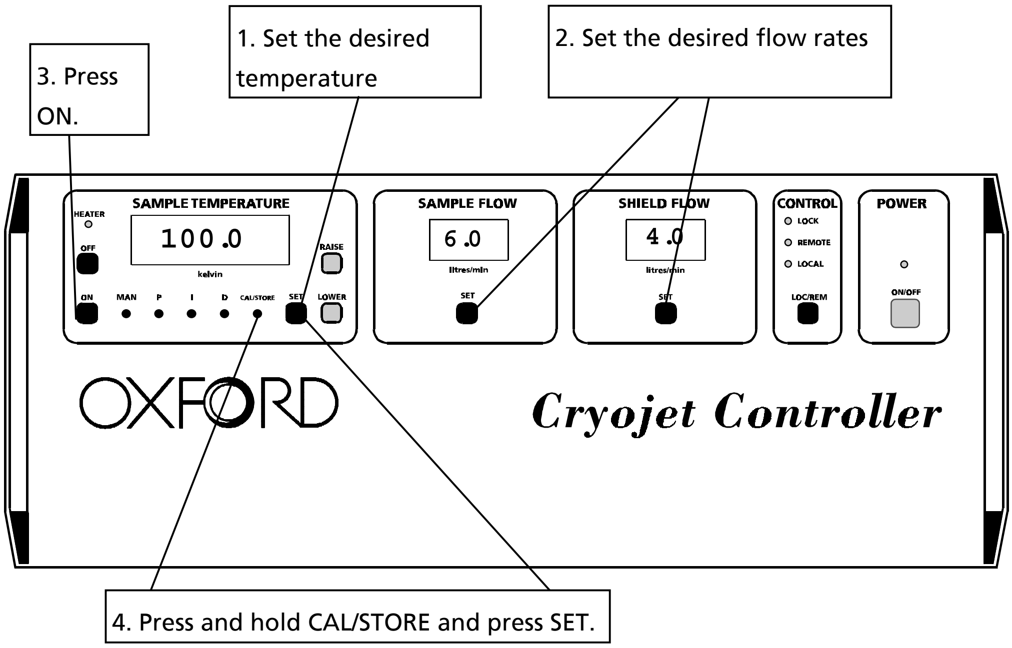

Figure 2 Procedure for putting controller into "Auto-start" mode

Start-up Condition	How to set this condition up
Non-Auto Start When Cryojet is switched on the gas flows are zero and automatic temperature control is OFF.	Set the temperature control off by pressing the OFF button on the left-hand side of the front panel. Use the STORE function: press and hold the recessed button CAL/STORE using a pointed object and press the SET button in the left-hand group of buttons. The letters "Stor" will appear on the display. (This will interrupt the flow momentarily). Now return the controller to Automatic temperature control mode for normal operation.
Auto-Start Sample flow and shield flow start as soon as Cryojet is turned on, with automatic temperature control. When the controller is switched on it will display the message "Auto" if it is in Auto-Start mode.	See Figure 2. Auto-Start is useful if there is a possibility of brief power failures when the Cryojet is running unattended. **Do not select Auto-Start unless you are sure that there will be liquid nitrogen in the dewar when the controller is switched on.**

2 Description of the system

The Cryojet is designed principally for X-ray diffraction studies. It allows a sample to be cooled or heated using a stream of gas without the need for windows between the X-ray source, sample and detector. Figure 3 illustrates the system.

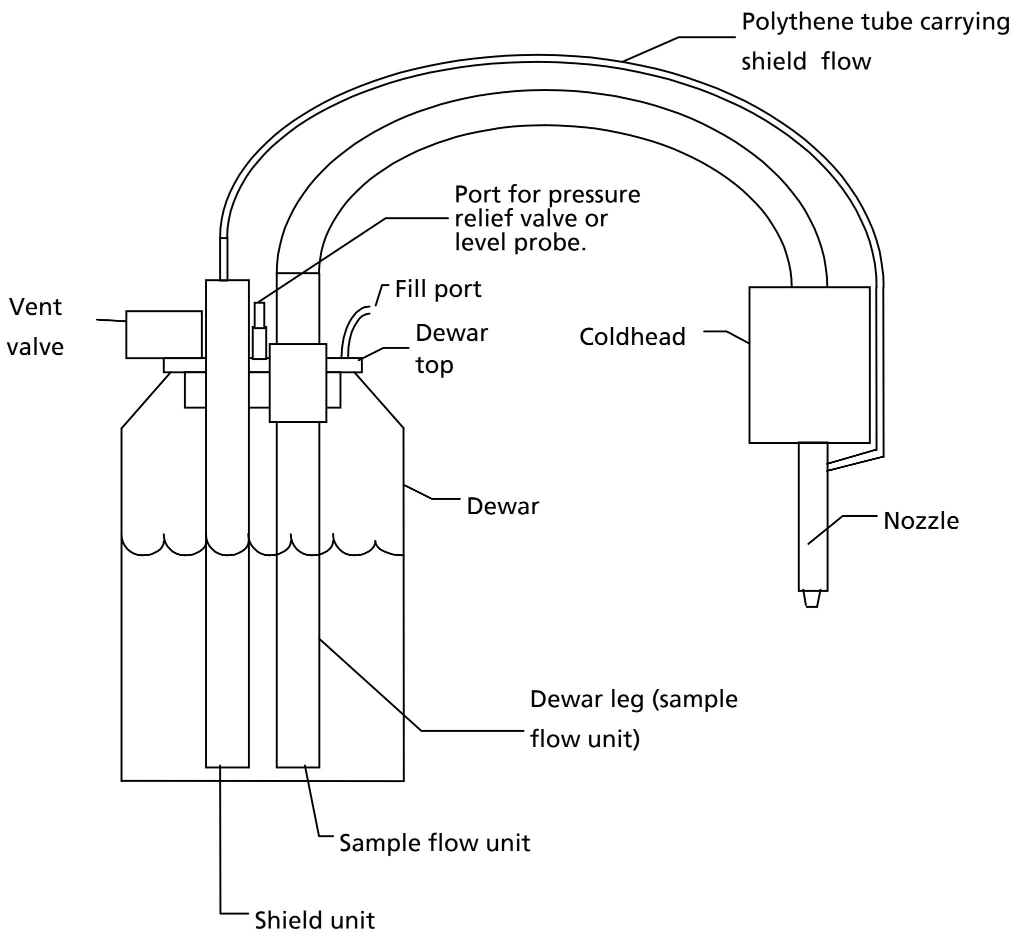

Figure 3 Sketch of Cryojet system

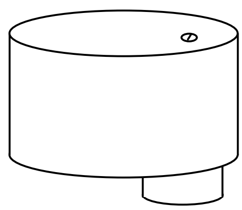

Figure 4 Vent valve

2.1 Sample flow unit

The dewar leg of the sample flow unit is immersed in the liquid nitrogen in the storage dewar. A heater at the bottom of the dewar leg boils nitrogen. The cold gas passes up the flexible stainless steel vacuum-insulated transfer tube into the coldhead. The coldhead contains a heat exchanger and heater. The gas then passes through the nozzle onto the crystal, forming the **sample flow**. The temperature of the sample flow can be regulated to any value between about 90 K and 300 K by the heat exchanger, using a temperature sensor in the nozzle.

A vacuum valve is situated at the top of the rigid section of the transfer tube, from which the Cryojet can be pumped to maintain the required thermal insulation.

2.2 Shield unit

The shield unit consists of a rigid vacuum-insulated tube that is inserted into the dewar. A heater at the bottom boils nitrogen. At the top of the unit there is a heat exchanger, with a heater and temperature sensor, which heats the nitrogen to room temperature. The nitrogen flows through the central stub of the shield unit to the coldhead via a length of polythene tubing. This **shield flow** then flows through the outer nozzle, preventing atmospheric water vapour from icing up the sample or the nozzle.

2.3 Stand

The Cryojet is supported via a stand that is adjustable to suit the geometry of the user's experiment.

2.4 Storage Dewar

A 75 l storage dewar is used to store the liquid nitrogen used for the sample flow and shield flow. The dewar has a top fitting with five ports as follows, shown in Figure 3.

- 51 mm hole: sample flow unit dewar leg
- 38 mm hole: shield unit
- Non-return vent valve
- 13 mm bent tube: for filling the dewar with liquid nitrogen
- stepped straight tube: for fitting level probe or pressure relief valve

2.5 Controller

The controller controls the gas flows and the temperature of the jet.

3 Unpacking and preparation

3.1 Unpacking the system

Carefully remove the Cryojet and all the accessories from the packing cases, and check the packing list to make sure that you have found all of the components. Examine the system to make sure that it has not been damaged since it left the factory. If you find any signs of damage please contact Oxford Instruments immediately.

A complete system consists of:

- Sample flow unit
- Stand with xyz adjustable stage
- Shield unit
- Dewar top fitting and 3 bolts
    - Includes fill port adaptor with flow restrictor
- For customers in the USA, 1/2" flare fitting
- Controller
- Polythene tubing (1.5 m)
- Electrical cables with the following labels:
    - coldhead
    - shield unit
    - dewar leg (and N2 level cut-off)
- Spares kit, including:
    - Allen keys (hexagonal wrenches): 8 mm, 6 mm
    - Tie-wraps
- Dipstick
- Liquid nitrogen storage dewar
- Pressure relief valve

The following components are options that will only have been supplied if ordered separately.

- Optional autofill system, or
- Liquid nitrogen hose
- Liquid nitrogen level meter and probe
- Pressurised liquid nitrogen supply dewar
- Dewar base with wheels
- High vacuum pumping system and lines

3.2 Preparing the system for operation

a) Check that the dewar top fitting is already fitted to the storage dewar, and is secured using the three M6 screws provided. Make sure that the three small screws that hold the top fitting together are loose. (The sample flow leg and shield leg will not slide into the dewar top fitting if these screws are tightened.) Remove the vent valve by pulling firmly upwards. (See Figure 4.)

b) Position the liquid nitrogen dewar about 1 metre from the intended position of the sample.

c) Set up the stand approximately in the intended experimental position. Slide the stage assembly onto the stand and clamp in position using the lever. (See Figure 6 and Figure 7 below.) Note that the lever can be rotated to any convenient position by pulling it to disengage the lever from the clamping mechanism.

d) Lower the sample flow unit dewar leg through the widest hole in the dewar top fitting to the bottom. (The wide part of the dewar leg is a tight fit through the O-rings in the top fitting.)

e) Slide the nozzle of the coldhead through the hole in the holder in the stand and secure using four caphead screws (supplied) at the base of the nozzle. Use the pre-existing bend of the flexible section when you arrange the coldhead in position. The shield tube running down the side of the coldhead must fit in the slot in the side of the holder. If the shield tube does not line up with the slot, rotate the entire dewar until it does.

f) Arrange the nozzle approximately in its intended operating position. After you have tilted the coldhead to the desired position, clamp the tilt adjuster in place using black knob. You find it convenient to use the 8 mm Allen key provided at the same time. To move the bracket assembly up or down the post smoothly, support the weight of the coldhead by putting your hand under the stage assembly near the coldhead.

g) Lower the shield unit into the next smaller hole, all the way to the bottom of the dewar. It is also a tight fit in the O-rings in the top fitting. Connect the fitting on the top to the fitting on the cold head using the polythene tube supplied. To connect, just push the polythene tube into the fitting until it stops.

h) Tighten the three small screws on the dewar top fitting.

i) Fit a suitable hose from your pressurised supply dewar to the Cryojet dewar. Some users use polythene tube. This becomes brittle when cold, and must be used with caution. Other plastics may not be safe, as they may be even more brittle. The fill port is fitted with a stainless steel coupling, equipped with a flow restrictor. This coupling has a 3/8" BSP parallel thread, with a 60° flare female sealing surface. If necessary, remove the top part of the coupling. Systems delivered in the USA also have an adaptor to convert this coupling to a 1/2" flare fitting (unless the system includes a nitrogen fill hose).

   If you have an Oxford Instruments autofill system, see section 8.

   If you have an Oxford Instruments nitrogen fill hose, fit it as follows. The brass adaptor fits onto your pressurised dewar, while the stainless steel adaptor fits onto the Cryojet dewar. Remove the brass adaptor from the hose, and screw it into the liquid outlet of your pressurised dewar, using PTFE tape. Screw the hose onto the brass adaptor using two spanners.

   Attach the other end of the nitrogen hose to the dewar top fitting, using the stainless steel compression fitting that is already fitted to the fill port. Slide the end of the hose onto the fill port, and tighten the nut securely using two spanners.

j) If you are using a level probe, first remove the screw in the side of the probe. Insert the probe through the stepped tube on the dewar top fitting. (Remove the pressure relief valve if one has been fitted.) Push down firmly until the level probe is as far down as it will go, and does not wobble. If desired, clamp the probe in position using the screw in the side.

k) If you are not using a level probe, make sure the pressure relief valve is pushed firmly in place on the dewar top fitting. (First remove the screw in the side of the valve. Fit the valve, and, if desired, clamp it in position using the screw in the side.)

l) Push the vent valve (Figure 4) firmly into position on the vent tube until it stops. This is a tight fit, as the vent valve is sealed by an o-ring onto the vent tube.

m) Fill the storage dewar. (If you do not fill the dewar to the top, make sure there is at least 10 cm of liquid nitrogen in it.) When the dewar is full, liquid nitrogen will spurt from the vent valve - stay well away. Replace the cap.

n) Before plugging in the controller, ensure that the voltage selector on the rear panel is correctly set for the intended supply voltage.

o) Connect the three cables. Just match the labels on the D-plugs with the labels on the back of the controller. The round connectors on the sample flow unit and shield unit are colour coded - match the colours when connecting. These connectors also have red dots - line up the red dot on the plug with the red dot on the socket.

p) Use the plastic tie-wraps to tidy up the system by attaching the tubes and cables together.

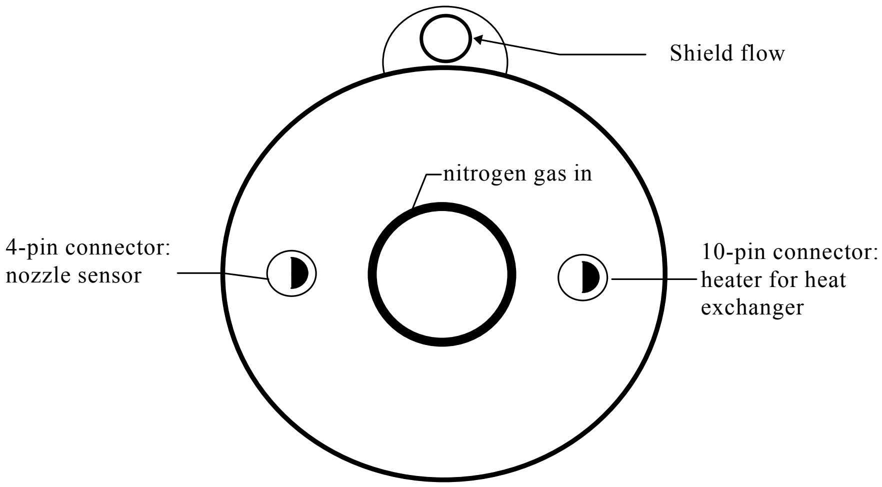

Figure 5 Schematic of coldhead top flange

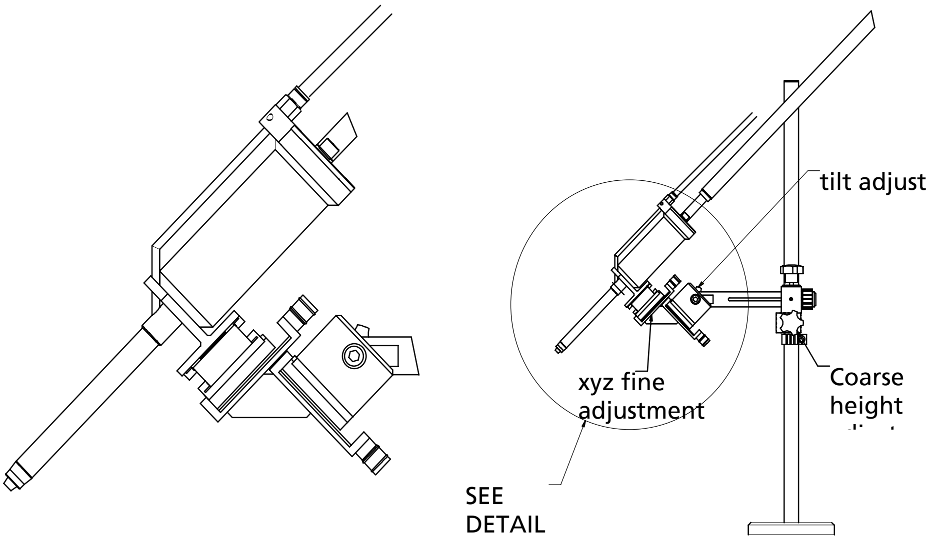

Figure 6 Coldhead and stand

(The appearance of the knobs may be different from that illustrated.)

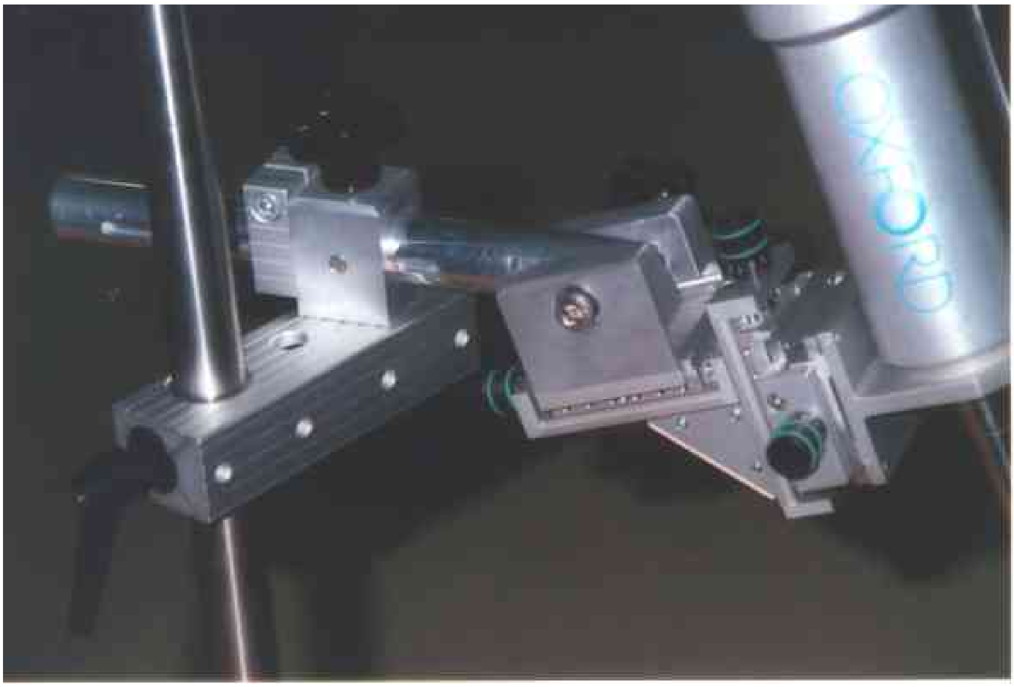

Figure 7 Close-up of stand assembly

4 Controller

Full details are in the Cryojet Controller Technical Handbook, included together with this manual. See also Figure 1.

4.1 Control of flow rates

The flow rates of the sample flow and shield flow are shown on the display in l/min (as if they were measured at atmospheric pressure and 293 K). To change the flow to any value from 0 to 10 l/min, hold down the SET button underneath the appropriate display and press RAISE or LOWER.

4.2 Control of jet temperature

There are three modes of operation: Automatic, Manual and Heat Exchanger Off. The light labelled “Heater” is on if the controller is in Automatic mode, or if it is Manual mode with non-zero voltage supplied to the heat exchanger.

Automatic

In this mode, the temperature of the jet is controlled automatically.

To put the controller into Automatic mode, press the ON button on the left-hand side of the front panel.

To display the Set Temperature press SET (left hand block of buttons) and keep it pressed.

To change the Set Temperature, hold down SET while using RAISE and LOWER.

To display the voltage being supplied to the heat exchanger at a given moment, press and hold the ON button on the left-hand side of the front panel.

To select Automatic mode, the sample flow must be at least 2l/min. If a flow has been selected but after a couple of minutes no flow can be felt, switch the heat exchanger **off** and see section 9.

Manual

In this mode, a fixed voltage is supplied to the heater in the heat exchanger. It is not recommended for routine operation.

To select Manual mode, press MAN. The default voltage is zero.

To display the voltage supplied to the heat exchanger, press and hold MAN.

To change the voltage supplied, press and hold MAN, and use RAISE and LOWER.

Heat Exchanger Off.

In this mode, no voltage is supplied to the heater in the heat exchanger. This mode will give the lowest possible temperature, but there will be some temperature variation.

To set this mode, press the OFF button on the left-hand side of the front panel.

4.3 Default Conditions on Start-up

There are two choices of start-up conditions, referred to as "Auto-Start" and "Non-Auto Start" as follows.

Non-Auto Start

- Gas flows equal zero;
  Heat exchanger is Off

To set these start-up conditions:
Set the heat exchanger off by pressing the OFF button on the left-hand side of the front panel.

Use the STORE function: press and hold the recessed button CAL/STORE using a pointed object and press the SET button in the left-hand group of buttons. The letters "Stor" will appear on the display. (NB This will interrupt the flow momentarily.)

Auto-Start

- Gas flows non-zero - can be configured to any desired value.
  Heat exchanger in Automatic mode

Auto-Start is useful if there is a possibility of brief power failures when the Cryojet is running unattended. **Do not select Auto-Start unless you are sure that there will be liquid nitrogen in the dewar when the controller is switched on.**

To set these start-up conditions use this procedure, illustrated in Figure 2:

1. Set the desired SET temperature.

2. Set the desired start-up gas flows.

3. Set Automatic mode by pressing the ON button on the left-hand side of the display.

4. Use the STORE function: press and hold the recessed button CAL/STORE using a pointed object and press the SET button in the left-hand group of buttons. The letters "Stor" will appear on the display. (This will interrupt the flow momentarily).

When the controller is switched on it will display the message "Auto" for a second or two if it is in Auto-Start mode.

4.4 Low Nitrogen Level Cut-out

A sensor attached to the sample flow unit dewar leg detects the presence or absence of liquid nitrogen. If the liquid level is below about 6 cm from the bottom of the dewar, all the heaters in the system will switch off automatically and the message "Lo N" will be displayed. To restore normal operation after you have refilled the dewar, you must switch the controller off and on again.

5 Operation

5.1 Suggested operating conditions

Always keep at least 10 cm of liquid in the dewar when the Cryojet is running, as lower levels could impair the performance. **Never allow the dewar to become empty when the Cryojet is running.**

Keep the plastic cap, the vent valve and the pressure relief valve (or level probe) in place on the dewar top fitting, to prevent water from the atmosphere entering.

A sample flow rate of about 6 l/min is recommended for most applications. (See 4.1 for how to set these flow rates.) The lower the flow rate, the higher the base temperature will be. Lower flow rates may be suitable if very low temperatures are not required, provided the sample can be placed very close to the nozzle. A flow rate of 4 - 5 l/min is usually sufficient for the shield flow - lower flow rates can be used if they are sufficient to stop the sample and nozzle icing up.

For a fast cool-down, set the sample flow rate to 10 l/min initially, and then reduce it to the desired value when the temperature is close to the set temperature.

5.2 Alignment of Nozzle and Use of Alignment Tip

The nozzle should be placed as close to the sample as possible to prevent icing. The limitation on this spacing depends on the diffraction angle required and the angle between the nozzle and the incoming x-ray beam. In almost every case the nozzle should be within 10 mm of the sample.

The jet should be centred on the sample as accurately as possible. One way to do this is to adjust the nozzle first so that the sample is in the plane of the nozzle. This makes it easier to centre the nozzle. Then move the nozzle back to the desired position using the micrometer stage. See Figure 8.

Another method is to use the Oxford Instruments alignment tip. Slide the device into the nozzle, and tighten the knob to clamp it in place. Place a sample mount with no sample on the goniometer. Adjust the nozzle so that the sharp tip is almost touching the place where the sample would be. Remove the sample mount. Loosen the knob and remove the alignment tip. Then place the sample on the goniometer. If required, move the coldhead forward using the micrometer.

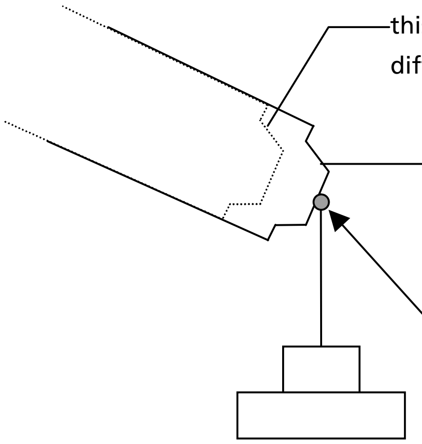

Figure 8 Possible alignment technique

5.3 Filling the dewar

The Cryojet can be run continuously for as long as required. The dewar can be refilled while the Cryojet is running.

Fit the liquid nitrogen hose from your supply dewar onto or into the fill tube. Make sure it is held in place so that it will not be blown free by the pressure of nitrogen during the fill. Use a low pressure in the supply dewar - preferably less than 1 bar. Open the valve on the supply dewar slowly, as the sample flow temperature may be temporarily affected by an increase in pressure in the dewar. Close the valve on the supply dewar slowly to stop the fill.

5.4 Using the dipstick to measure the level of liquid nitrogen

A dipstick (a long plastic rod) has been provided as a simple way of estimating the level of liquid in the dewar. (A digital level meter and probe are also available - see separate manual if these have been supplied.) Slowly lower the dipstick into the dewar through the fill port until it hits the bottom, and wait a couple of seconds. Then pull it out, and wait a few seconds for frost to form on the surface. The length of the frosted section is the depth of liquid in the dewar. Do not touch the cold part of the dipstick with your bare hands.

For depths between 10 cm and 80 cm, the dewar holds about 0.98 litres for every additional centimetre of liquid. (See calibration chart below.) A flow of 1 litre/min of gas corresponds to a consumption of 0.086 litre/hour of liquid, i.e. about 0.088 cm/hour. In addition, there is a boiloff from the dewar of about 0.10 cm/hour

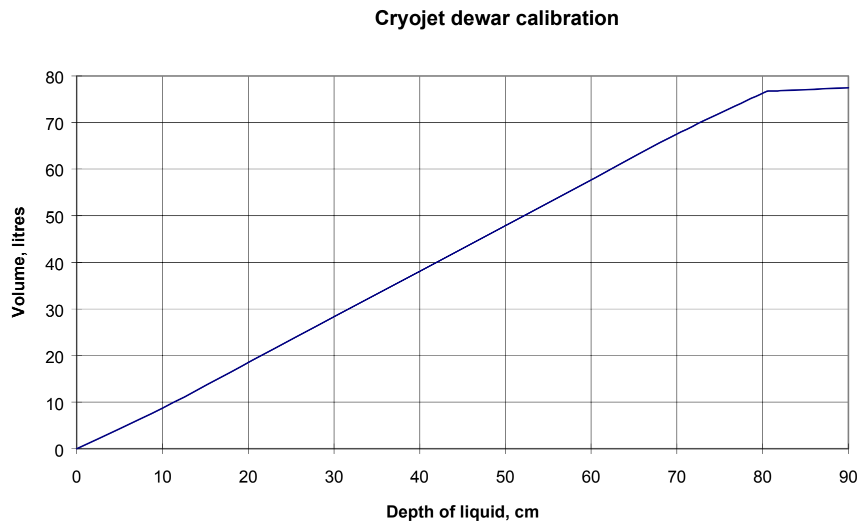

Figure 9 Cryojet dewar calibration

5.5 Warming up the system

The system should be switched off whenever it is not in use. To prevent the remote possibility of ice or water building up in the coldhead (which is designed to prevent this happening), *either*

a) Block the inner nozzle with a rubber stopper. The cold head can then be left to warm up naturally.

Or

b) Set the temperature to 300 K and allow the displayed temperature to reach room temperature before switching off. To speed this up, reduce the sample flow to about 3 l/min.

If you do not use the Cryojet for several days, the dewar can warm up. It is then possible for water to collect at the bottom. This could block the system when it freezes, although the dewar legs have been designed to prevent this. This can be prevented in several ways:

(a) Before allowing the dewar to warm up, raise both dewar legs. To do this, loosen the three small screws in the dewar top fitting. Raise the shield unit and the sample flow unit dewar leg by a centimetre or two, then clamp them in place by tightening the three small screws. Leave them in place when you later refill the dewar. (To maximise the available time between refills, you may want to lower the legs to the bottom later, but it is easier to do this when the dewar top fitting is no longer cold from the filling process.); or

(b) Before refilling a warm dewar, attach a dry air or nitrogen supply to the outer nozzle. Gas will then flow backwards through both the sample flow unit and the shield flow unit, blowing out any water from the holes at the bottom.

If either the sample flow dewar leg or the shield unit is removed from the dewar, then before putting it back check that the small holes in the bottom are clear of ice, using a stiff wire such as a paper-clip.

It is not possible to remove the shield unit from the dewar top fitting when it is cold. To dismantle the system when cold, remove the entire system including the dewar top fitting from the dewar in one unit.

5.6 Evacuating the Outer Vacuum Chamber (OVC)

The OVC of both the sample flow unit and shield unit have to be pumped to high vacuum to make sure that they provide the required thermal insulation.

Both are pumped in the factory, but this needs to be repeated occasionally. When the system is new, all of the materials inside the vacuum space are likely to outgas quickly, and this will affect the quality of the vacuum. This does not mean that the system is leaking.

The OVC of the sample flow unit should be pumped if either of the following symptoms appear:

- The sample flow unit feels cold to the touch, or condensation or frost appears. Some condensation or frost is normal on and just above the dewar top fitting.
- The jet does not reach the desired temperature, or the voltage supplied to the heat exchanger heater is lower than that given for the relevant temperature in the test results appended to this manual.

If the Cryojet has not been used for a month or more and the dewar has warmed up, it is often advisable to pump the vacuum in the sample flow unit before starting to use it again.

The OVC of the shield unit should be pumped out whenever water or ice condenses on the outside of the rigid dewar leg. Some condensation is normal on and just above the dewar top fitting.

Both OVCs contain a sorb at the bottom of the dewar leg. Ideally they should be pumped overnight, and during pumping the dewar leg should be heated to about 100 ºC to make sure that the sorb outgasses. You can do this by setting the appropriate flow rate to 8 l/min (not more) with the dewar leg out of the dewar. You must support the dewar leg so that the bottom end is not touching anything that could melt. Do **not** insulate it in any way, as this could cause overheating. Before you set the flow rate you must over-ride the low nitrogen level cutoff by removing the plug from the socket labelled "Low nitrogen level cutoff" on the back of the controller. Remember to replace the plug afterwards.

Do not bake the flexible transfer tube or coldhead as these contain plastic components.

It is also possible to pump the OVC while the Cryojet is operating, although the vacuum will not last as long in this case, as the sorb has not been outgassed.

Pumping with a two-stage rotary pump that has a base pressure of about 10^{-3} mbar is sufficient. A diffusion pump or turbo pump would also be suitable.

5.7 Calibration of Sample Temperature

Precise estimation of the temperature of a real sample in the cold jet is not straightforward, as it will be affected by the size and location of the sample, the orientation of the nozzle, and by heat conduction along the sample support. Contact Oxford Instruments for calibration information.

5.8 Stability

The temperature displayed is normally stable to about □0.1 K. Small fluctuations in temperature will occur when the dewar is refilled.

5.9 Icing of Crystal Support and Goniometer

The shield flow is designed to prevent icing of the sample and nozzle. A slight build-up of snow is likely on the crystal support and goniometer. In most cases no action need be taken, provided that the incoming beam can be collimated to exclude any ice on the crystal support. Increasing the shield flow rate to 10 l/min for a minute or so may help to remove frost from the crystal support.

6 Control from a computer

The controller can also be controlled from a computer using the RS232 interface or the optional GPIB (IEEE-488) interface. In either case, you can use the ObjectBench software provided. An installation manual is provided. Windows 95 or 98 is required. This section explains the basics of how to control the Cryojet. Full information on topics such as plotting parameters against time, saving data to a file, and creating macro programs is available through the on-line help.

A unique feature of the Cryojet controller and other Oxford Instruments products is the ability to connect a number of instruments simultaneously, to a single RS232 port on a computer, and to control each one independently. This is done by means of an Isobus cable which carries a single “master” connector (25-way D socket) and up to eight, daisy-chained “slave” connectors (25-way D plugs). This is available from Oxford Instruments Cryospares.

6.1 RS232 Connection Hardware

The bi-directional serial data link from the computer is connected via a 25 way D-socket on the rear panel. The controller is configured as a DCE with the standard pin outs given below. The majority of computer RS232 interfaces are configured as a DTE and are fitted with a 25 way D plug. For this type of connector, a simple lead connecting pin 1 to pin 1, pin 2 to pin 2 and so on is all that is required. For computers fitted with a 9 way D plug for RS232, (AT style COM port), a standard "AT lead" fitted with a 9 way socket and a 25 way plug is required.

If for any reason you plug the computer into a different electricity supply circuit from the Cryojet controller, you should use an Oxford Instruments ISOBUS cable to ensure electrical isolation.

6.2 GPIB (IEEE-488) Connection Hardware

If the optional GPIB interface is fitted, connections to the GPIB are made via a standard 24 way GPIB cable, conforming to the standard IEEE-488.1. Connections should be made using a standard GPIB cable. If you wish to connect both a Cryojet controller and a level meter to a GPIB port on a computer, use a “GPIB Gateway” - contact your sales engineer or Oxford Instruments Cryospares.

Note: **GPIB connections should never be made or broken whilst the controller or any of the instruments connected to the Bus are powered up. Failure to observe this precaution can result in damage to one or more instruments.**

Never plug the computer into a different electricity supply circuit from the Cryojet controller.

6.3 Control Using the Oxford Instruments ObjectBench software package

ObjectBench is provided with the Cryojet. This section describes how to install and run the Cryojet driver in ObjectBench. For other information, see the ObjectBench manual.

6.3.1 Installation of Cryojet driver in ObjectBench

Window in which command is found	**Command to be selected, or information to be entered**	**Result**
Main ObjectBench	Configure	Configure Submenu appears
Configure submenu	Add/remove instruments	Configure Instruments window appears
Configure Instruments	Add	Load instrument drivers window appears
Load instrument drivers	Select driver "cryojet.drv". Change the configuration name, or leave it as "default" if only one Cryojet is installed.	Cryojet driver window appears, resembling the physical controller front panel.
Configure Instruments	OK	

6.3.2 Setting up the Cryojet driver

The basic commands that must be used when setting up the instrument driver are as follows.

Window or location in which command is found	**Command to be selected, or information to be entered**	**Result**
Cryojet driver	Configure	Configure submenu appears
Cryojet driver, Configure submenu	Interface	
Cryojet driver, Configure Interface	Select the type of communication link and the COM port you are using on your computer. This will be 1 if your computer only has one COM port (serial port). Set the ISOBUS (or GPIB) address you wish to use for the Cryojet controller. (You may leave the default value of 1, providing no other instrument on the ISOBUS at the same time uses the same address)	Configure Interface window appears
Cryojet driver, Configure interface	Set address. Then follow the instructions on the screen.	
Physical Cryojet controller front panel	Press and hold CAL/STORE, and press SET (NB This will interrupt the flow momentarily.)	The address is stored in the EEPROM, so it is not lost when the controller is switched off.
Cryojet driver	Connect	Driver becomes "live", current nozzle temperature is displayed.

6.3.3 Control of the Cryojet

The Cryojet instrument driver in ObjectBench is intended to be self-explanatory.

Control of the instrument may either be LOCAL from the front panel, or REMOTE via the computer interface. The LOC/REM button may be used to switch between LOCAL and REMOTE.

When LOCK is lit, the instrument is locked into either local or remote control and the LOC/REM button has no effect. At power up, the controller is locked in LOCAL.

When the controller is in REMOTE, many of the front panel controls are disabled. Those controls that only affect the display will still work, but those that could change the operation of the instrument will not. If LOCK is lit whilst in REMOTE, all the front panel controls are inoperative.

Note that there is no indication on the instrument driver as to whether the controller is in LOCAL or REMOTE. If the controller is in LOCAL, it will not respond to computer commands. To rectify this, use the DISCONNECT and then the CONNECT command in the instrument driver. This will put the controller into REMOTE.

6.4 Restoring Standard Calibration Parameters

If any of the set-up parameters are changed by mistake, or the EEPROM (non-volatile memory) becomes corrupted, the controller can be restored to the state in which it left the factory, by using the back-up disc provided, and ObjectBench software. Use the following sequence of operations and menu selections.

This feature is not available from the Cryojet instrument driver. Instead, you must add the instrument driver called ITC503.drv (using Configure / Add/remove instrument / Add).

DISCONNECT the Cryojet driver if it is connected.

If the controller ISOBUS address is not the default value (1), or you are using a COM port (serial port) other than COM1 on your computer, set up the ITC503 driver to the correct address and COM port using Configure / Interface.

Press and hold the Shift key while clicking CONNECT in the ITC503 driver. 0.000 will appear in the driver display.

In the ITC503 driver window, select SETUP/MEMORY.

LOAD ->

Enter the filename of the back-up file: a:*nnnnn*.ram, where *nnnnn* is the Oxford Instruments Project Number of your Cryojet.

OPEN

PUT->

OK

Then STORE the restored data in the controller EEPROM. (Press and hold CAL/STORE and press LOC/REM. NB This will interrupt the flow momentarily.)

DISCONNECT

If you want, remove the ITC503 driver (using Configure / Add/remove instrument).

7 Setting of P, I and D Control Terms

The PROPORTIONAL, INTEGRAL and DERIVATIVE control terms may be displayed and set by means of the recessed P, I and D buttons. Use a pointed object to press these buttons. The system is already set with optimised values of these parameters: it should **not** be necessary to change them, or to read the rest of this section. The default values are P=20, I=2.5, D=0.5

P indicates the PROPORTIONAL BAND in Kelvin to a resolution of 0.001K

I indicates the INTEGRAL ACTION TIME in minutes, covering a range of 0 to 140 minutes in steps of 0.1 minute.

D indicates the DERIVATIVE ACTION TIME in minutes, covering a range of 0 to 273 minutes (Though values beyond 70 minutes are unlikely to be required in practice.).

In North America, a different terminology exists for 3-term control.

PROPORTIONAL BAND is replaced by its reciprocal, GAIN.

INTEGRAL ACTION is replaced by RESET. This may either be specified as a time (as for integral action) or as its reciprocal, "REPEATS PER MINUTE".

DERIVATIVE ACTION is replaced by RATE. Again this may be specified as a time or as repeats per minute.

RAISE and LOWER may be used to vary the control terms whilst in LOCAL control. When you have changed the values, carry out a STORE command as follows: Press and hold the recessed button CAL/STORE, and press SET. This will store the new values in non-volatile memory. (NB This will interrupt the flow momentarily. If a STORE command is executed when the controller is in Automatic mode, then this will also set the “Auto-Start” mode - see 4.3.)

The main purpose of DERIVATIVE action is to reduce overshoot, when approaching a new set temperature. For most systems derivative action will not be required and may be left at zero. (Hold LOWER pressed for a second after 000.0 is displayed to ensure that there is not a small residual setting of less than 0.05 mins which will show as zero).

The P and I controls should not normally be set to zero, since this would correspond to ON/OFF control.

The following procedure gives a good rule-of-thumb for setting the controls to a value that is close to optimum.

a) Set I for a time much longer than the expected response time of the system.

b) Set D to zero.

c) Select AUTO and reduce P until the temperature starts to oscillate above and below some mean value (not necessarily the set point).

d) Time the period of oscillation (in minutes). This is a measure of the response time of the system.

e) Set I to a value approximately equal to the response time. Then increase the P setting to a point where oscillation just ceases. Note the value of P at this point, then set it to approximately double this value. This gives a good starting point for the P and I control terms.

f) Test how the system responds to step changes in the SET point and modify the P and I settings for a reasonably fast response without excessive overshoot.

g) If overshoot remains a problem following a large step change in SET, try the effect of adding some DERIVATIVE action. A good initial setting is half to one third of the system response time measured above. This will probably require P to be re-optimised for best results.

h) When optimising P, I and D the aim should be to achieve the lowest values of all three terms, consistent with no oscillation and an acceptably small amount of overshoot. This will give the fastest response for the system.

When adjusting the control terms remember that reducing P increases the controller gain. This can cause some confusion when the concept of PID control is first encountered.

8 Liquid Nitrogen Autofill System

The optional autofill system automatically refills the 75 litre unpressurised dewar from a pressurised supply dewar.

Warning: **There is always a possibility that this autofill system will fail, resulting in the entire contents of your pressurised dewar being spilt on the floor. You MUST have a contingency plan so that you can deal with this safely. An autofill system is not likely to be safe in a small or medium sized room, unless you use safety devices such as oxygen level alarms and self-contained breathing equipment. The autofill system is recommended for use with portable pressurised dewars (of 160 litres or less), not with large nitrogen tanks.**

Warning: **A pressure relief valve has been fitted to the supply pipework on the autofill to safely discharge any pressure generated by liquid nitrogen trapping. This valve should NOT be removed without consulting an expert.**

The autofill system consists of the following components.

- Nitrogen level probe
- Level probe cable
- Nitrogen hose with solenoid valve
- Nitrogen level meter with IEC power cable.

The pressurised supply dewar is normally provided by the customer.

The level meter has its own detailed manual, but this section describes the basic operation as part of an autofill system. The level meter has already been set up to function correctly in the 75 litre Cryojet dewar. The display shows the depth of liquid, expressed as a percentage of the maximum depth, which is 805 mm for the standard 75 litre Cryojet dewar. Do not run the Cryojet below 13% depth (10 cm), as this will impair the performance.

The level meter is set up in the factory with the following default settings.

FULL	95%	Filling stops at this level
FILL	15%	Filling starts at this level
LOW	10%	Warning light appears on level meter.

Each level meter is set up for the level probe supplied with it. If you use a different probe, or you use the probe in a different dewar, re-calibrate the level meter as described in its manual.

8.1 Setting Up the Autofill System

Your pressurised dewar must be equipped with a pressure regulator or pressure relief valve which will ensure that the pressure does not exceed 1.5 bar. If the dewar has a pressure build circuit, you may not need to use it, as adequate pressure will build up naturally. A pressure in the range 0.3 to 1 bar (4 to 15 psi; 30 to 100 kPa) is recommended, but up to 1.5 bar (22 psi, 150 kPa) is acceptable.

Insert the level probe into the straight stepped port on the dewar top fitting (Figure 3). Push it down firmly until it goes no further. You may need to twist it to ensure the O-ring seal slides over the top of the port.

Connect the level probe to the level meter using the cable supplied.

Check that the level meter is set for the correct supply voltage. This is indicated on the back panel, just above the power supply socket. Switch on. Check that the label on the solenoid valve indicates the correct voltage.

Remove fitting A from nut B on the autofill assembly (Figure 10).

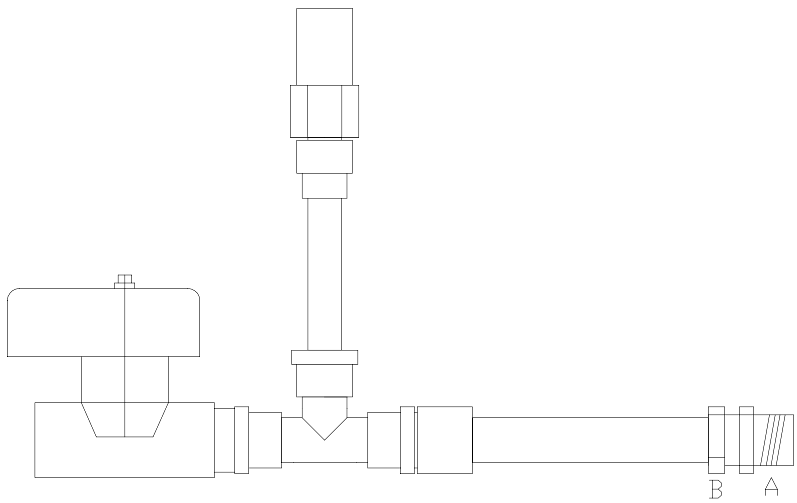

Figure 10 Autofill nitrogen hose, detail of the end near the pressurised dewar

Screw fitting A into the liquid outlet of your pressurised dewar, using PTFE tape. (The appearance of item A will depend on your dewar).

Engage the brass tube into fitting A, ensuring the ferrule is correctly positioned on the tube and tighten nut B using two spanners. It is good practice for the solenoid valve to be above the hose, so that water does not drip onto it.

Attach the other end of the nitrogen hose to the dewar top fitting, using the stainless steel compression fitting that is normally already fitted to the end of the hose. If the nut and ferrule (olive) are already on the dewar top fitting, just slide the end of the hose onto the fill port, and tighten the nut securely using two spanners. If not, slide the nut and the ferrule onto the fill port first, then slide the end of the hose on afterwards until it stops. Ensure the hose is pushed down as far as it will go while you tighten the nut.

Plug the IEC electrical power socket wired into the level meter into the solenoid valve.

The Cryojet will not give maximum stability unless the pressure in the dewar remains very close to atmospheric pressure. The autofill system is supplied with a restriction in the fill hose to prevent the pressure in the dewar rising. If you observe an unacceptable fluctuation in temperature when the fill starts, then set the ball valve on your supply dewar only partly open, to restrict the flow of liquid and gas when the solenoid valve opens. If the temperature fluctuations are negligible but the fill is unacceptably slow, then you may wish to remove or reduce the restriction. To do this, remove the fill hose from the dewar, and unscrew the stainless steel compression fitting from the end of the hose. The restrictor is a plastic plug with three holes, glued inside this fitting. You can either drill a larger hole in this plug, or request a new plug with no restrictor from your Oxford Instruments sales representative.

Note: This will increase the temperature fluctuation caused by filling.

The autofill is ready for use. If the Cryojet dewar is below the FILL level, it will start to fill.

8.2 The Solenoid Valve

The valve is closed when no power is supplied to it. When the solenoid valve is energised (open) for a long period it will become very hot. This is a safe operating temperature.

Normal valve operation is indicated by a sharp click. Sluggish operation indicates that cleaning of the core assembly is required.

The valve can be opened by plugging it directly into the mains electricity supply, using a suitable cable. The hose can then be used without the level meter, to transfer nitrogen under manual control.

9 Fault finding

Symptom	Diagnosis and suggestions
"Lo N" appears on the controller	There is less than 6 cm of liquid in the dewar. Do not operate the Cryojet with less than 10 cm of liquid. Refill the dewar, then switch the controller off and on again.
"Hot 1" appears on the controller	The nozzle temperature is above the hot limit, set by default to 320 K. The Cryojet is only specified to operate up to 300 K. Reduce the set temperature, and switch the controller off and on again. Alternatively, the nozzle sensor is faulty, or the cable is not connected.
Controller will not stay in Auto mode (Heater light will not stay on)	Sample flow must be at least 2 l/min.
RAISE, LOWER and Heater On/Off do not work	Controller is set to REMOTE. Press LOC/REM to restore to LOCAL control (unless controller is LOCKED in REMOTE mode).
The controller does not respond to computer control.	The controller may be in LOCAL. If it is, use the DISCONNECT and then the CONNECT command in the instrument driver. This will put the controller into REMOTE.

The temperature stays at room temperature, even though the sample flow is not set to zero.	Check that cable from the controller to the coldhead is plugged in correctly, and that there is liquid nitrogen in the dewar. The heat exchanger could be blocked with moisture from the atmosphere, if it was switched off while cold. Set the shield flow to zero, and the sample flow to 10 l/min. Use the back of your hand to feel whether there is any flow through the inner nozzle. (1) If there is no flow at all, and the temperature does not start to fall, remove the whole assembly from the dewar (sample flow unit, shield flow unit and dewar top fitting), and purge it for at least two hours by flowing dry gas through the nozzle. (If you attach the gas supply to the outer nozzle, disconnect the shield flow polythene tube and block the shield flow inlet to the coldhead to prevent the gas escaping.) Alternatively let the sample flow unit warm up nearly to room temperature, and then purge it with air. (2) If you can feel some flow with your hand at 10 l/min sample flow and 0 l/min shield flow, and the temperature does drop a little, set the heat exchanger to 300 K, with the sample flow at 10 l/min. Purge the coldhead in this way for at least an hour. For the first few minutes, check continually that there is some flow through the inner nozzle, as the heat exchanger could overheat otherwise. If method (2) does not work, the sample flow dewar leg could be blocked. Use method (1). See 0.
Water or ice condenses on the sample flow unit or the shield unit	Pump the vacuum (section 4.3). When the dewar is refilled, the nitrogen gas venting from the dewar will cool the dewar legs. This is normal, and does not require action. If the vacuum valve becomes too cold, however, the O-ring will not seal effectively, and the vacuum will be lost. If this occurs, repump the vacuum, and take steps to direct the vented nitrogen away from the vacuum valves.
Cryostat OVC cannot be pumped to high vacuum	Check the OVC for leaks using a mass spectrometer leak detector if available. If there is no leak there may be too much moisture in the OVC and it should be pumped with a rotary pump with the gas ballast valve open.

Cryostat will not reach its specified base temperature	Check that there is at least 10 cm of liquid in the storage dewar. Move the dewar so that the curve of the flexible transfer tube is as gentle as possible - avoid sharp bends. It may help to tilt the coldhead closer to the horizontal. Pump the vacuum in the OVC.
Polythene tube carrying shield flow develops heavy condensation or frost.	Check that the shield flow rate responds to changes in the flow rate set using the controller. If the actual shield flow is high even when it is set to 0 l/min, the controller is at fault. See also below "Suspected fault in a heater or sensor".
The controller fails to operate:	Check fuses as follows. The main electricity supply plug is fused in some countries. The fuse for the controller is located behind the voltage indicator on the back panel.
Ice forms on the nozzle	Check that the shield flow is sufficient, and that the polythene tube is connected. The shield unit could be blocked. Remove entire system from dewar, and warm it to room temperature. Check with a wire or paper clip that the holes at the base of the shield unit and sample flow unit dewar leg are clear. Dry the system, and replace it in the dewar. To prevent recurrence, see 0.
Suspected fault in a heater or sensor	See section 10.

10 Wiring Information

To measure the resistances of the heaters and sensors, the easiest way is to disconnect the cables from the controller, and measure between the pins of the D-plugs.

The 15 way D-sockets on the controller labelled “Cryojet dewar leg” and “Shield unit” are **identical**. You can monitor voltage outputs to the coldhead while the jet is running by disconnecting the shield unit from the controller (or *vice versa*), and connecting a voltmeter across the pins of the socket you have disconnected.

10.1 Shield Unit

Component	Pins on 15 way D-connector	Pins on round connector fixed to shield leg	Typical resistance between pins, (when disconnected from controller), ohms	Typical output of controller measured using the spare socket “Cryojet dewar leg”, when shield leg is plugged in
Boiloff heater	2 and 10	7 and 8	35 - 40	up to about 40 volts, depending on flow
Heat exchanger heater	3 and 11	5 and 6	35 - 40	up to about 40 V, fluctuating
Platinum sensor	5 and 13 (or 5 and 14)	1 and 2 (or 1 and 4)	about 105 ohms at room temp, falling to 20 ohms at 70 K	pins 5 and 14: 100 mV at room temperature
Leads for platinum sensor	13 and 14	2 and 4	about 1 ohm	

10.2 Sample flow unit: 15 way D-connector (labelled "Cryojet dewar leg")

Component	Pins on 15 way D-connector	Pins on 4-pin round connector fixed to dewar leg of sample flow unit	Typical resistance between pins, (when disconnected from controller), ohms	Typical output of controller measured using the spare socket "Shield unit", when sample flow unit is plugged in
Boiloff heater	1 and 9	1 and 2	35 - 40	up to about 40 volts, proportional to the square root of the flow

10.3 Sample flow unit 9-way D-connector (labelled "Coldhead")

Component	Pins on 9 way D-connector	Pins on round (Fischer) connectors fixed to coldhead	Typical resistance between pins, (when disconnected from controller),	Typical output of controller measured when D-plug is removed
Heat exchanger heater	6 and 7	1 and 2 of 10-pin connector	35 - 40 ohms	usually 0 - 2 V
Platinum sensor, voltage leads	1 and 2	1 and 2 of 4-pin connector	about 105 ohms at room temp, falling to 20 ohms at 70 K	undefined
Platinum sensor, current leads	4 and 5	3 and 4 of 4-pin connector	about 105 ohms at room temp, falling to 20 ohms at 70 K	typically 20 to 30 V
Leads for platinum sensor	1 and 4 (and 2 and 5)	1 and 3 (and 2 and 4) of 4-pin connector	about 1 ohm	undefined